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3. The plant employed about 2000 persons, many of whom were of German origin. Some of these employees commuted to work from as far away as forty kilometers. Because of this, new three story apartment dwellings with twenty-four apartments in each for the plant workers had been started [redacted].

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[redacted] Each apartment had a living room, kitchen, and two bedrooms.

4. In addition to a period [redacted] (course of instruction), the technical nature of which was subordinated to the political aspects, the workers received two to three hours of political indoctrination each week. A Polish translation of a technical booklet for engineers was issued to each worker who in turn had to memorize the entire contents whether or not he understood those contents. In nearby Katowice, a city of 80,000 population, was a large library of technical publications translated into the Russian language. Second-hand German technical books were available but were expensive.

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6. A detailed description of the phenol process, by stages, is contained in the following paragraphs.

#### A. Chlorobenzene Stage

Benzene from storage was passed through a drier packed with solid caustic (NaOH), thence to a lead-lined chlorinator packed with iron Raschig rings. The actual feed line entered above the middle of the chlorinator and passed through the packed region to a point near the bottom, thus allowing a short pre-heating period for the benzene. The metered chlorine was fed into the bottom of the reactor and controlled to give a 25-30 % chlorination, thereby minimizing the production of di- and higher-chlorinated benzenes. Initially, the chlorinator was heated with process steam to the desired temperature and thereafter the exothermic reaction was controlled by river water cooling of the jacket. The chlorination mixture was sent to a stripping column in which a benzene cut (80°C) was separated from the monochlorobenzene (MCB) and recycled to the chlorinator with sufficient benzene for process make-up. From the stripper, the crude monochlorobenzene distilling at 132°C, was transferred to a rectifying column where it was purified and then sent to storage. The bottoms were being discarded in October 1952. More than the required amount of monochlorobenzene needed for the phenol process was being produced because of the ready market for it in Poland. The capacity of the monochlorobenzene unit could easily be stepped up by chlorinating to a higher degree but in so doing a larger cut of higher chlorobenzenes would be obtained. Hydrochloric acid resulting from the reaction was piped to an absorber where it was reclaimed as aqueous acid for use later in the process. This absorber was a vertical Pyrex column packed with graphite and employing concurrent flow. The hydrochloric acid stripped from the benzene chlorinator with the aid of 6°C refrigeration at the column head. No difficulties had arisen concerning corrosion because the process at this stage was anhydrous. [redacted] tantalum equipment does not exist in Poland nor is it readily available in the rest of Europe.

#### B. Monochlorobenzene Conversion to Phenol Stage.

In this stage, 60% caustic was diluted to 20% strength using process condensate in a power-agitated mixing vessel to which was added 10% diphenyl ether to suppress the side reaction by which diphenyl ether is formed. In a molar ratio of two sodium hydroxide to one monochlorobenzene, the components were fed to the high pressure reactors through a high pressure proportioning

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pump. The feed rate of this pump was controlled by the speed of the drive motor while one of the piston strokes could be varied during operation. This variable stroke piston controlled the feed rate of the monochlorobenzene. The mixed feed at 300 atmospheres pressure next passed through a high pressure heat exchanger heated by the countercurrent flow of the completed reaction mixture. The three high pressure reactors were well lagged and only the first two were electrically heated to about 400° C, the third being a holder to afford sufficient time to complete the reaction. After initial cooling in the high pressure heat exchanger, the mixture consisting of sodium phenolate, diphenyl ether, salt, and water with a small amount of un-reacted monochlorobenzene was further cooled to below 100° C before it was admitted to the expansion valve. This pressure reducer was of the homogenizer type with a rotating cone impinger. The mixture next went to a combination condenser-separator where the diphenyl ether supplied to and formed in the reaction was decanted continuously and sent to storage. The excess diphenyl ether was being sold for use as a heat transfer medium in Poland. The phenolate liquor was then conducted to the neutralizer.

#### C. Neutralization of the Phenolate Liquor.

The liquor from the previous stage plus the aqueous hydrochloric acid obtained from the absorber were fed to a vessel equipped with a powered agitator to free the phenol from its sodium salt. The resulting liquor separated into two distinct phases which were then withdrawn with the aid of a sight glass and valves to their respective storage containers. The phenolated brine was in the lower phase while the aqueous phenol was in the upper phase.

#### D. Phenol Purification.

The phenol liquor was transferred to a stripping column in which the phenol was separated from any residual monochlorobenzene and water through the use of a 92°C ternary azeotrope composed of phenol-water-monochlorobenzene. This azeotrope was condensed and separated. The upper phase was returned to process condensate for re-use and the lower phase was returned to a suitable point on the column. The phenol component was withdrawn from the still-pot and piped to a rectifying column where it was distilled at atmospheric pressure. The final product had the following characteristics:

(182° C fraction)

Required.....freezing point 39.6 - 41.0°C

Actual.....freezing point 40.4 - 40.5°C

Colorless, soluble in water without turbidity

The bottoms were being discarded.

#### E. Phenolated Brine Treatment.

The brine, containing about 2 % phenol, was sent to a stripping column. Here a binary water-phenol azeotrope containing 11 % phenol was obtained, which on condensation separated into two phases. The lighter phase consisted of 6 % aqueous phenol while the lower phase consisted of about 50 % phenol. The 6 % liquor was returned to process condensate for re-use and the 50 % phase was combined with the raw phenol feed to the phenol stripping column. By this process of recycling, the phenol losses from the process were held to an absolute minimum. The stripped brine, although being discarded, will ultimately be concentrated in double effect evaporators after passing through regenerative active carbon purifiers. The resulting concentrated brine will supply the electrolytic cells with which it is intended to produce the necessary caustic and chlorine for the entire process. In October 1952, however, no facility existed in the plant for producing electrolytic caustic. Requirements had to be fulfilled from the electrolytic caustic plant at Moscow.

#### F. Miscellaneous Notes on Equipment and Processing.

When the solid caustic drier was exhausted, the spent caustic was made up to the required 60% concentration and held in storage for the hydrolysis step.

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The iron for the iron-catalyzed chlorination was furnished by the iron Raschig ring packing. Heat for the chlorinator and the two monochlorobenzene columns was supplied from process steam at about 225 psi. The Pyrex for the hydrochloric acid absorber was obtained from England. The phenol purification columns and the brine stripper were all heated by the use of circulated hot water held under suitable pressure to obtain temperatures of 200 - 250°C.

25X1 [ ] this was an innovation of the Krebs Company and that no particular difficulties had been experienced in its use. The feed for the system was made up by use of process steam condensate which was considered to be economical and safe and to have desirable heat transfer characteristics. The activated carbon absorbers were reactivated with steam as needed. Two units were available in each line. The proportioning pump was obtained from the Corblin Company, Paris. It was a three piston unit giving the desired pressure and proportioning simultaneously. The speed varied from 60 - 130 rpm. with only the monochlorobenzene piston having the variable stroke. The pressure release by means of a blow-out disc was set for 340 atmospheres. The pressure reducer was obtained from Bergdorfer Eisenwerk, Bremen. A long range objective was to obtain free phenol in the hydrolysis step through the use of higher temperatures and less caustic. No progress had been made on this point by October 1952.

7. The total plant capacity was designed for 300 metric tons per month, allowing five days each month for maintenance and repair. The installation consisted of two complete units in parallel, only one of which was in operation (half the rated output by October 1952) pending training of suitable operators to handle both units simultaneously. 25X1 [ ] the level of competence of the available operators was so low that damage to the plant through mishandling, especially in the high pressure installations could be expected. The ultimate use of the phenol was for phenol-formaldehyde type resins and synthetic fibers. A small amount of it was to be used for medical purposes because of its high purity. Some shipments of phenol, consigned to the Warsaw area, were noted. Three Krebs employees still supervised production and the training of personnel for the phenol unit in October 1952.

8. The development of synthetic fibers in Poland was said to be far behind that of other countries. Plans had been purchased from the Soviets for methanol, acetaldehyde and acetic acid plants, the equipment for which was to be manufactured in Poland. Certain techniques available in Poland were said not to be up to the demands and specifications of the chemical industry. For example, 25X1 [ ] the copper lining on the pressure vessels in another part of the plant blistered considerably and even peeled away from the base metal. Bearings in Polish-built compressors gave particular difficulty since no bronzes were to be had, only white metals and babbitts. However, good lubricating oils were available. 25X1 [ ] the data on the refrigerating-unit lubricants as being a turbine oil #3 with high viscosity at low temperature, 50° Engler value at 50°C and 14° Engler value at room temperature, and a 28°C freezing point.

25X1 [ ] COMMENT:

- 1 Although the production of phenol as described in this report is conventional in most respects, the process has been reported in detail because several novel features were noted.

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